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INTERNAL FRICTION MEASUREMENT IN HIGH PURITY
TUNGSTEN SINGLE CRYSTAL

by Georges E. Rieu
Lewis Research Center
Cleveland, Ohio 44135
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George E. Rieu[*]

Lewis Research Center

ABSTRACT

Internal friction peaks observed after small deformation ($\epsilon \simeq 2.5$ %) in high purity tungsten single crystals (R/R > 50 000), between liquid helium temperature and 800° K in the frequency range 30-50kHz, are studied as a function of orientation.

An orientation effect is observed in the internal friction spectra due to the creation of internal stresses.

The elementary processes related to these peaks are discussed in terms of kink generation and geometric kink motion on screw and edge dislocations in an internal stress field.

INTRODUCTION

In recent years there have been several attempts to differenciate the respective contribution of screw and edge dislocations to the internal friction spectrum induced by deformation in B.C.C. metals (refs 1, 2, and 3). However, the presence of impurities in these materials reduced the difference between screw and edge dislocation mobility, induced impurity-dislocation interactions, and caused the pinning of the dislocations at high temperature, generally making difficult any interpretation of intrinsic effects. In addition, the effect of grain orientation in the deformation did not permit a precise determination of the amount of deformation, particularly in the preyield range.

In the present work, we tried to control these two major experimental problems by using tungsten single crystals preoriented and free

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[*]E.N.S.M.A. University of Poitiers, France
Visiting National Research Council Associate.

of impurities in solution. Tungsten was chosen for this study because it is the only BCC transition metal presently able to meet the high purity requirement. Single crystal samples, preoriented in the three principal orientations were used in order to determine the orientation dependence.

To maintain the initial purity of the samples during the experiments, we used an internal friction apparatus within an ultra-high vacuum chamber, capable of high temperature operation to study in-situ recovery.

EXPERIMENTAL

In the experiments we used tungsten single crystal rods 6.35 mm (0.25 inches) in diameter and 50 mm (2 inches) in length, oriented [100], [110], and [111] along the axis, within $\pm 2^{o}$ of the specific orientation. All the specimens were zone refined and had a resistivity ratio greater than 50,000 which correspond to a total point defect concentration less than 1 atomic ppm (ref. 4). A polycrystalline rod of 99.995 percent tungsten was used for comparison.

We deformed the sample at room temperature in compression using an Instron machine, at a strain rate of 0.01/min. Guides of annealed copper were used to prevent bending of the samples without introducing any significant radial stresses (fig. 1).

The internal friction measurements were made at about 45 kHz between 30 and 800 K, using longitudinal vibration. At high temperatures, measurement was made with an electrostatic drive, frequency modulated for detector of the oscillation (ref. 5). This permits measurement at higher temperatures and the possibility of in-situ recovery; for low temperature measurements, separate electrodes for the electrostatic drive and for detection were used. In this case, the detection system employed an electrostatic amplification; the same sample holder was used in both cases in order to avoid errors due to manipulating. This mounting achieves precision in the measurements approaching 10^{-7} in Q^{-1}. These two mountings are shown in Figure 2. The low temperature measurements can be made either during cooling or warming of the sample, using the low temperature chamber and sample mounting represented in Figure 3; A lock-in-amplifier in the detection circuit eliminates any noise produced by the filling of the chambers with liquid

nitrogen or liquid helium.

A schematic diagram of the electronic systems used is shown in Figure 4.

RESULTS

Annealed samples

To eliminate any residual stresses due to polishing or manipulating, we annealed all of the samples 2 hours at 1900° K under a vacuum of about 2×10^{-9} torrs.

The internal friction spectrum observed after annealing is shown in Figure 5. As with polycrystalline molybdenum (ref. 1), our polycrystalline tungsten shows a very low damping over the entire temperature range $(Q^{-1} < 4 \times 10^{-6})$. It is associated with a linear variation of modulus with temperature. In contrast, the high purity single crystal specimens show damping (or peaks) as high as 3×10^{-4}, and exhibit several modulus defects.

The [111] crystals show two small peaks at low temperature at around 170° K, and a broad composite peak over the temperature range $300-700^O$ K. The corresponding modulus defect variation is perturbated between $450-600^O$ K and decreases rapidly above 600° K. This faster decrease corresponds exactly to the high temperature fall-off of the broad peak.

Similar high temperature behavior is shown by the [100] crystal but smaller in amplitude; however, no peak is detected at low temperature.

Besides the two small low temperature peaks, the [110] crystal shows two distinct peaks at 320° K and 460° K. The 320° K peak is slightly affected by temperature. Cooling to liquid nitrogen temperature makes it higher ($\times 1.15$) and shifts it about 15° K toward lower temperatures. Heating to 475° K makes it lower ($\times 0.8$) and shifts it about 12° K toward higher temperatures. After aging by heating to 730° K at a rate of 0.5 K/min, it almost disappears ($\times 0.08$). The 460° K "peak" is too narrow to be a relaxation peak and looks more like a "phase transformation" spike. It does not correspond to any significant change in the modulus.

After 2.5 percent deformation and 2 hours annealing at 1800° K under a 2×10^{-9} torr vacuum, these two peaks disappeared and a broad, temperature stable peak appeared. The disappearance of the 320° K peak and the linear variation of the corresponding modulus suggest that this peak may be a more stable configuration of the 320° K peak.

Deformed samples

The samples were deformed in compression at room temperature. The corresponding stress-strain curves for the "corner" orientations and for the polycrystalline material are given in Figure 6. The [100] and [111] crystals exhibited a very low proportional limit which we associate with the high purity of the samples. They yielded smoothly and showed significant strain hardening. The [110] crystals showed sharper yields at higher stress and exhibted less work hardening. Similar stress-strain behavior has been observed previously in tension for less pure single crystals (refs. 6 and 7). In contrast, polycrystalline samples yielded very sharply at higher stresses, showing no strain hardening until more than 1 percent deformation.

The internal friction curves obtained after 2.5 percent compression at room temperature (Fig. 7) show several distinct peaks: a small low temperature peak, around 70° K, a large double peak at about 170 and 220° K, a broad complex peak, particularly strong in the [110] crystal between 200° K and 500° K, and a high temperature peak around 800°K. All of these peaks appear for all the samples, except the high temperature peak for the polycrystalline specimen.

The 70° K peak which appears clearly for every corner orientation and for the polycrystal can perhaps be associated with the α peak observed in Nb by Chambers (ref. 8). The corresponding modulus defect (Fig. 8) is about twice the maximum damping height (subtracting the 170° K peak contribution). We thus assume that it corresponds to a single relaxation process. From the temperature at which it appears we estimate it to have an activation energy of about 0.1 eV.

The 170° K and 200° K peaks correspond respectively to the α and β peaks reported by Chambers (ref. 9) and Secretan (ref. 10), and to the α composite peak reported by Muss and Towsend (ref. 11). We cannot

separate the respective contribution of each one of these peaks to the modulus defect (Fig. 8), but the magnitude of this effect indicates a complex relaxation process. Their activation energies are 0.21 eV and 0.66 eV respectively.

The high temperature side of this double peak is obscured by the 200-500° K broad complex peak, particularly for the [110] crystal. This complex peak is affected by temperature. It increases and shows a more defined maximum around 320° K after heating to 400° K at 0.5K/min ([110] crystal). It is almost eliminated by heating to 720° K ([100] crystal) revealing the 170-200° K double peak and the corresponding modulus defect. .The 800° K peak is presumed to be the same peak observed by Chambers (ref. 12) (γ peak), Martinet (ref. 13) and Schnitzel (ref. 14). In fact, its temperature for this frequency is remarkably consistent with Martinet and Schnitzel's results. This temperature correspond to an activation energy of 1.5 eV. An important inverse and irreversible modulus defect occurs at the same time which shows a saturation 50 degrees lower than the maximum of the peak. We notice also in figure 7 a small perturbation occuring in the internal friction and modulus for all samples at the temperature of the spike.

DISCUSSION

On first examination of these results, we note a significant difference between the spectrum of the polycrystalline sample and that for high purity single crystal, either annealed or after deformation. Initially, therefore, we will try to interpret these results on tungsten independent of any previous results on molybdenum or niobium, and we will even cautiously compare these results with other tungsten results.

The two series of peaks observed after annealing and deformation correspond to different structures. After annealing, we can assume that the crystals contain some dislocations and vacancies formed by thermal stresses during the cooling, and by the local plastic deformation occurring around the support points of the sample. The existence of active dislocations is confirmed by the presence of the 170° K and 220° K peaks on the [111] crystal.

The peaks observed at high temperature after annealing have the same shape of a "dislocation background" with a decrease of internal friction occurring between 600° K and 700° K. This temperature region corresponds to stage III recovery of W. The smaller damping observed during the cooling suggest a pinning of the active dislocations. However, the more rapid decrease of the modulus for this temperature range rejects this interpretation.

In contrast, this pinning effect is suggested by the modulus variation after deformation, an one can think of a vacancy migration toward the dislocations, . The 800° K peak would then be associated in this case with a vacancy-dislocation interaction occurring just prior the pinning of the dislocations by migrating impurities. Martinet (ref. 13) studied the recovery of this peak and concluded the occurence of this kind of interaction. However, the 100° K difference between the migration temperature of these defects (600-700° K) and the temperature of the maximum of this peak (800° K), and also the saturation observed in the variation of the modulus, make vacancy-dislocation interaction arguments unacceptable.

In fact, electron microscopy observation after a few percent deformation (2 percent) at room temperature (ref. 15) shows clearly that the dislocations substructures are only long screws dislocations parallel to the [111] direction. This suggest their active participation in the process responsible for these peaks; the ratio of screw dislocations to non-screw dislocations is too large to associate any large peak with non screw dislocations; we will suggest therefore an interpretation in agreement with Seeger-Šesták interpretation for W (ref. 3). In essence then, the 70° K peak must be the expected peak corresponding to the formation of double kinks in edge dislocations, and the 170° K peak may correspond to the motion of kinks in screws dislocations.

For the [110] crystal, Rose et al (ref. 6) pointed out the fact that the only two operating slip directions lie in a plane whose normal is perpendicular to the deformation axis. Therefore, dislocation motion will require high stress. Accordingly, we can expect this sample to have more discontinuities on the screws dislocations and have large internal residual stresses. But, Stephens (ref. 15), Arsenault and Lawley (ref. 16) noticed that the dislocation mobility is sensitive to the internal stresses. We can therefore interpret the broad 200-500° K peak by the motion of kinks

under stress fields that can include different defects (jogs, vacancies, dislocations and self interstitials) acting over a few atomic diameters. These effects are less important on [111] and [100] crystal because for these orientations more slip directions are active. The dislocation motion will be easier during deformation and the crystal will contain therefore less internal residual stresses.

The 800° K peak may be due to the formation of double kinks in screw dislocations. In this case, we can interpret the modulus defect by a possible readjustment during the double kink formation toward a more stable geometric kink configuration.

These interpretations of the $200-500^O$ K and 800° K peaks will explain particularly:

(1) The increase of the $200-500^O$ K peak and $170-220^O$ K peaks after heating, because the corresponding stress release allows an easier motion of kinks.

(2) The diminution of the $170-220^O$ K peak after heating to 720° K because of the decrease in the number of geometric kinks.

(3) The $200-500^O$ K peak diminution or disappearence after heating to 720° K for the preceding reason and also because of the large stress release.

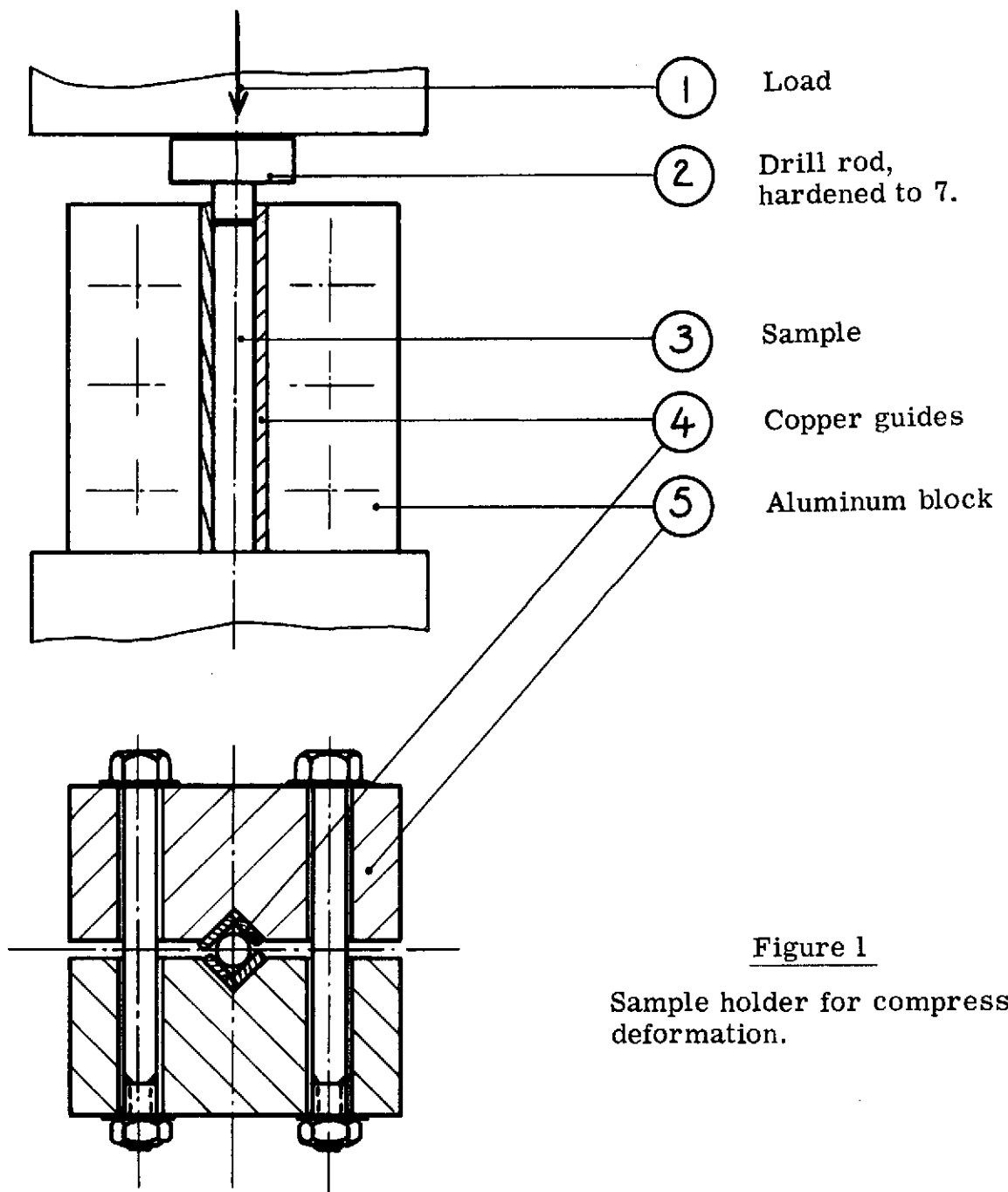(4) The saturation of the modulus defect after reaching a stable geometric kink configuration.

(5) The correspondance between the height of the $170-220^O$ K peak and the magnitude of the inverse modulus defect. Both are directly related to the number of geometric kinks.
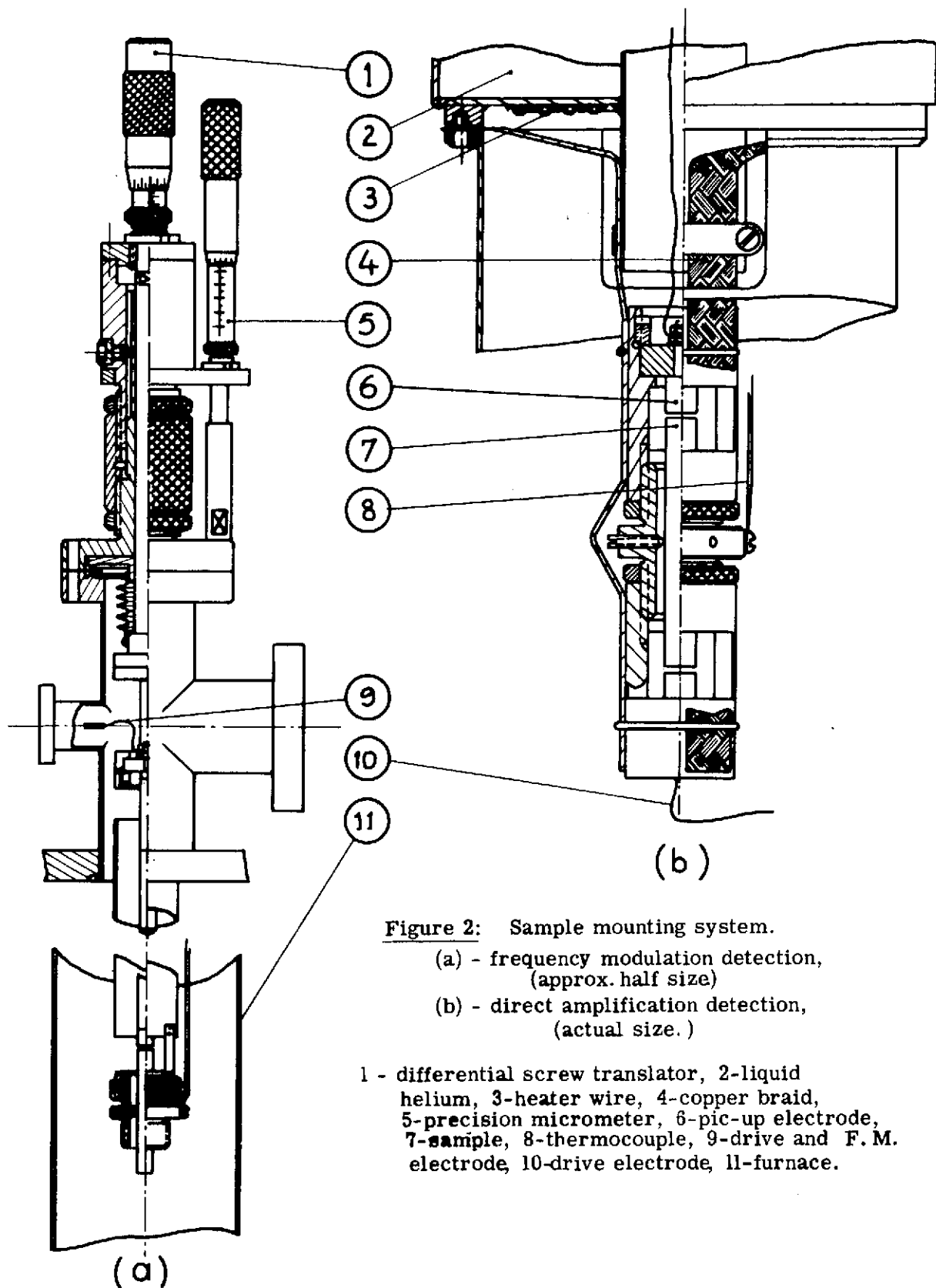
It is also useful to compare these results with those observed for Nb and particularly with those obtained for polycrystalline Mo (ref. 1). The modulus behavior is exactly the same after deformation; in particular, we observe the same fast increase of the modulus at a temperature corresponding to the stage III recovery of these metals; in Mo this modulus increase is associated with a sharp fall-off of the internal friction, corresponding to a pinning; but this same modulus increase in W does not correspond to any variation in the damping. So it is hazardous at best to associate these peaks for the different metals until additional experimental evidence is available.

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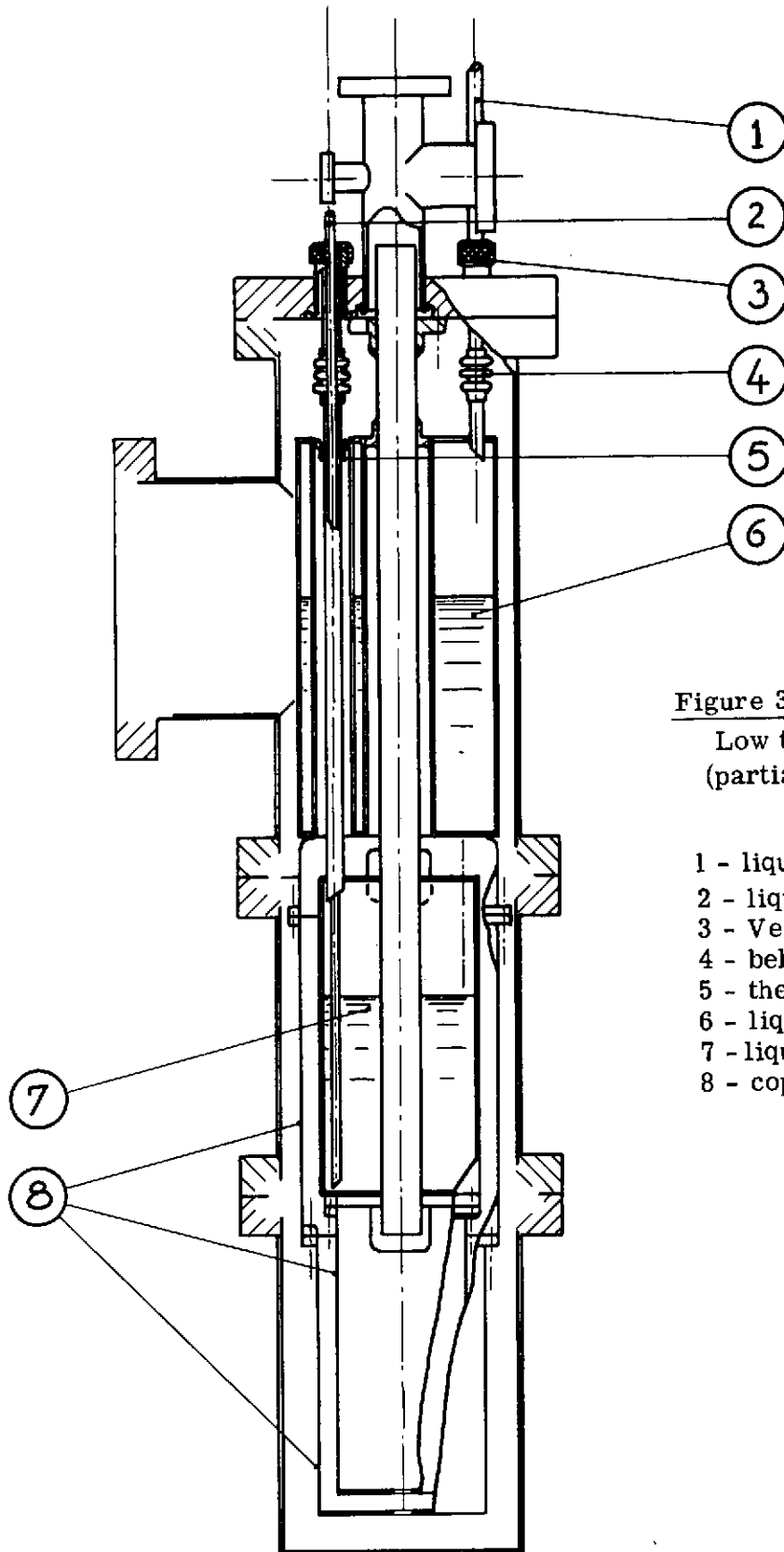
①	Load
②	Drill rod, hardened to 7.
③	Sample
④	Copper guides
⑤	Aluminum block

Figure 1

Sample holder for compressive deformation.

Figure 2: Sample mounting system.
  (a) - frequency modulation detection,
       (approx. half size)
  (b) - direct amplification detection,
       (actual size.)

1 - differential screw translator, 2-liquid
  helium, 3-heater wire, 4-copper braid,
  5-precision micrometer, 6-pic-up electrode,
  7-sample, 8-thermocouple, 9-drive and F. M.
  electrode, 10-drive electrode, 11-furnace.

Figure 3:
  Low temperature chamber
  (partial representation).

1 - liquid nitrogen feedthrough
2 - liquid helium feedthrough
3 - Veeco feedthrough
4 - bellows
5 - thermal contacts
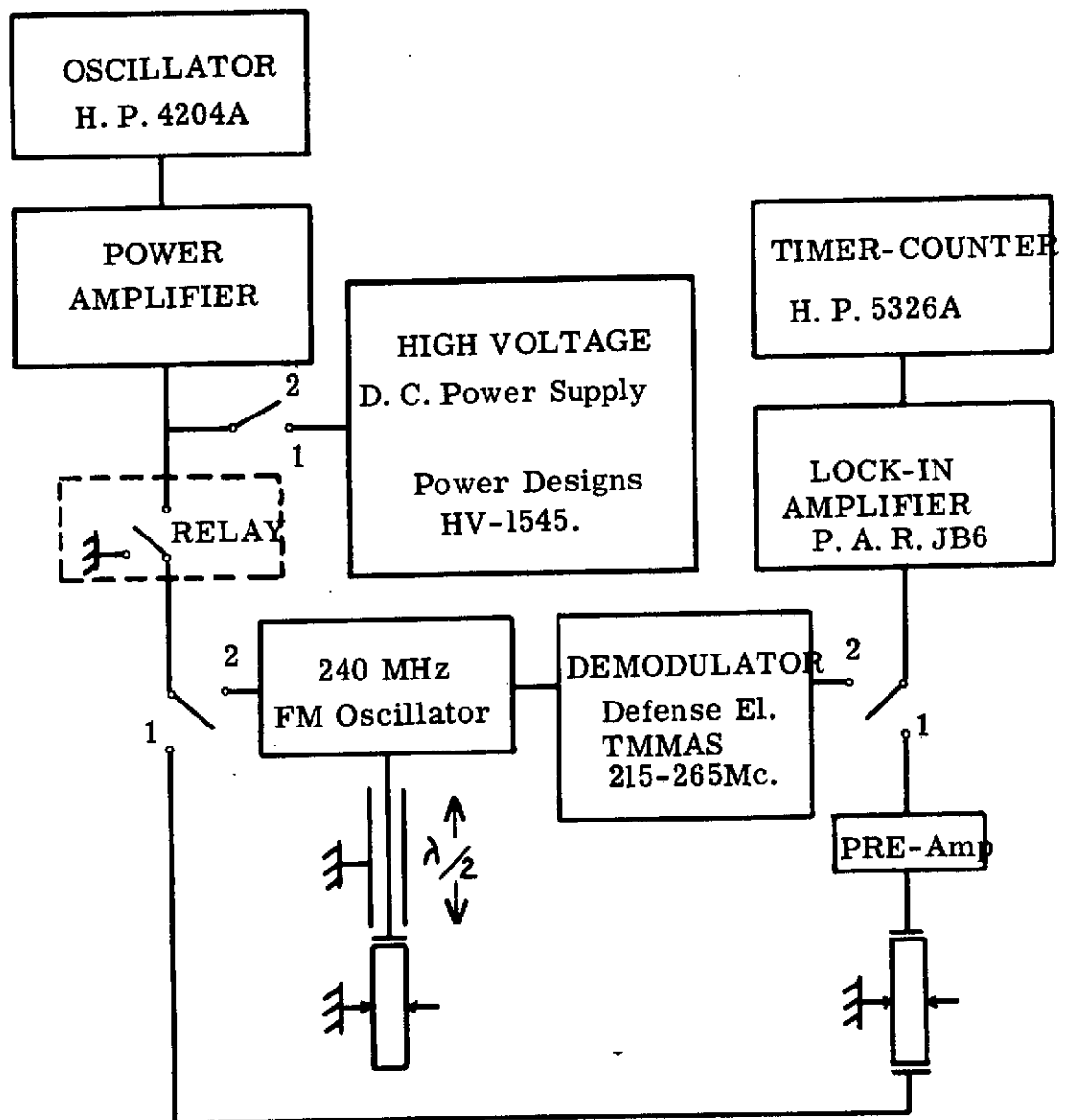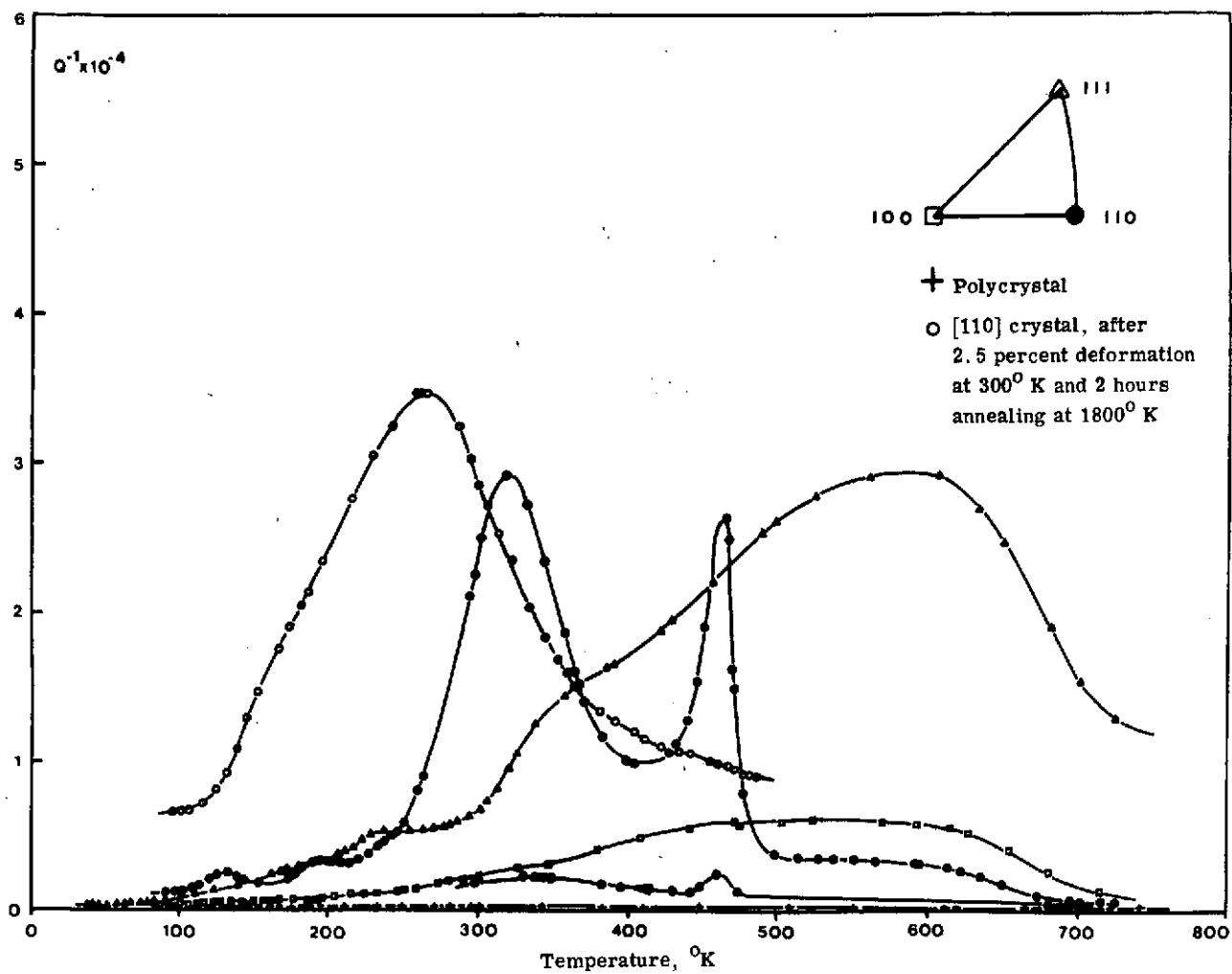6 - liquid nitrogen
7 - liquid helium
8 - copper shields

OSCILLATOR
H. P. 4204A

POWER
AMPLIFIER

2

1

RELAY

1

HIGH VOLTAGE
D. C. Power Supply

Power Designs
HV-1545.

TIMER-COUNTER

H. P. 5326A

LOCK-IN
AMPLIFIER
P. A. R. JB6

2

240 MHz
FM Oscillator

DEMODULATOR 2

Defense El.
TMMAS
215-265Mc.

1

1

$\lambda/2$
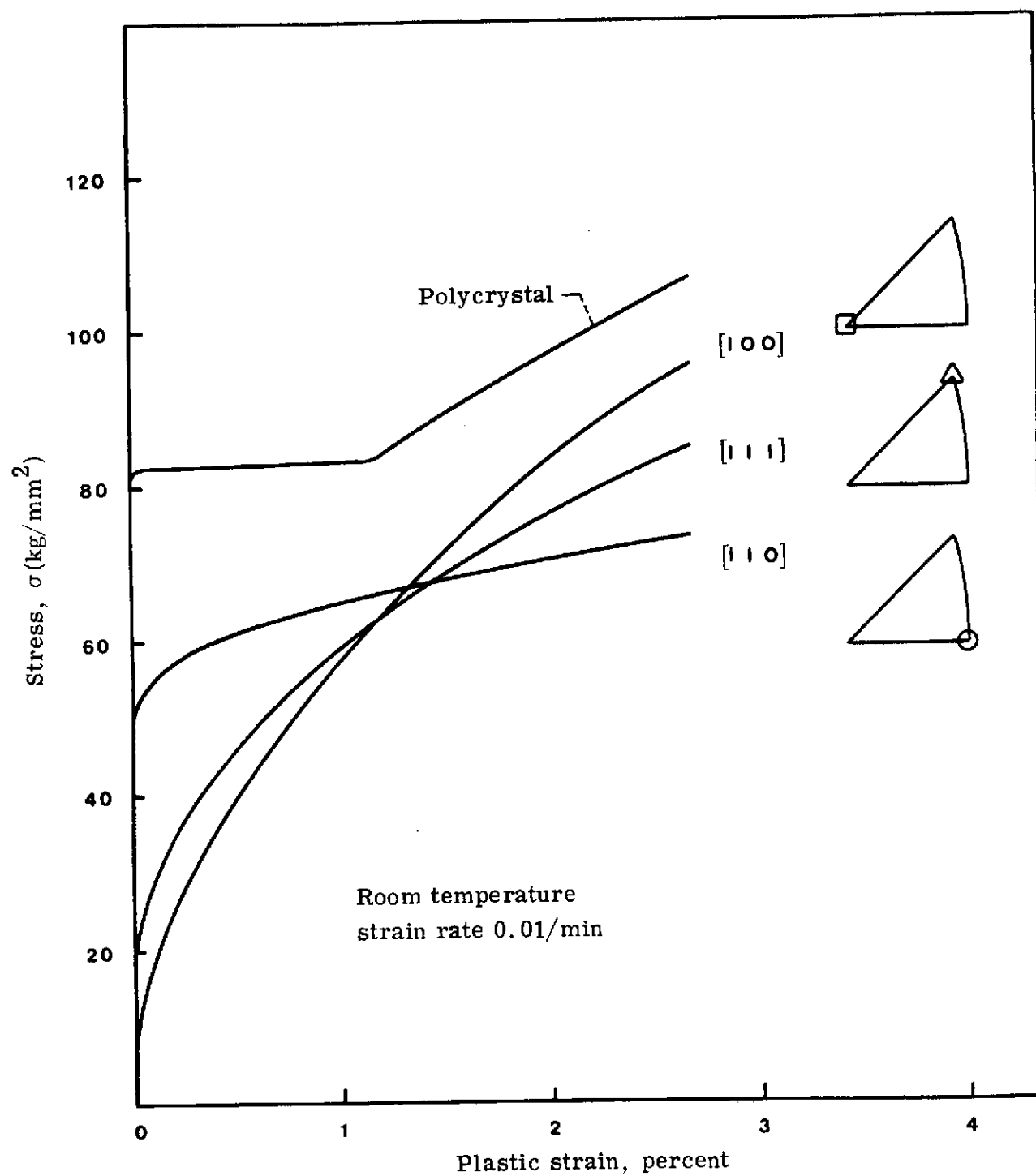
PRE-Amp

Figure 4: Electronic equipment

Figure 5: Annealed samples.

Figure 6: Representative stress-strain curves for tungsten single crystal and tungsten polysrystal deformed in compression at room temperature.
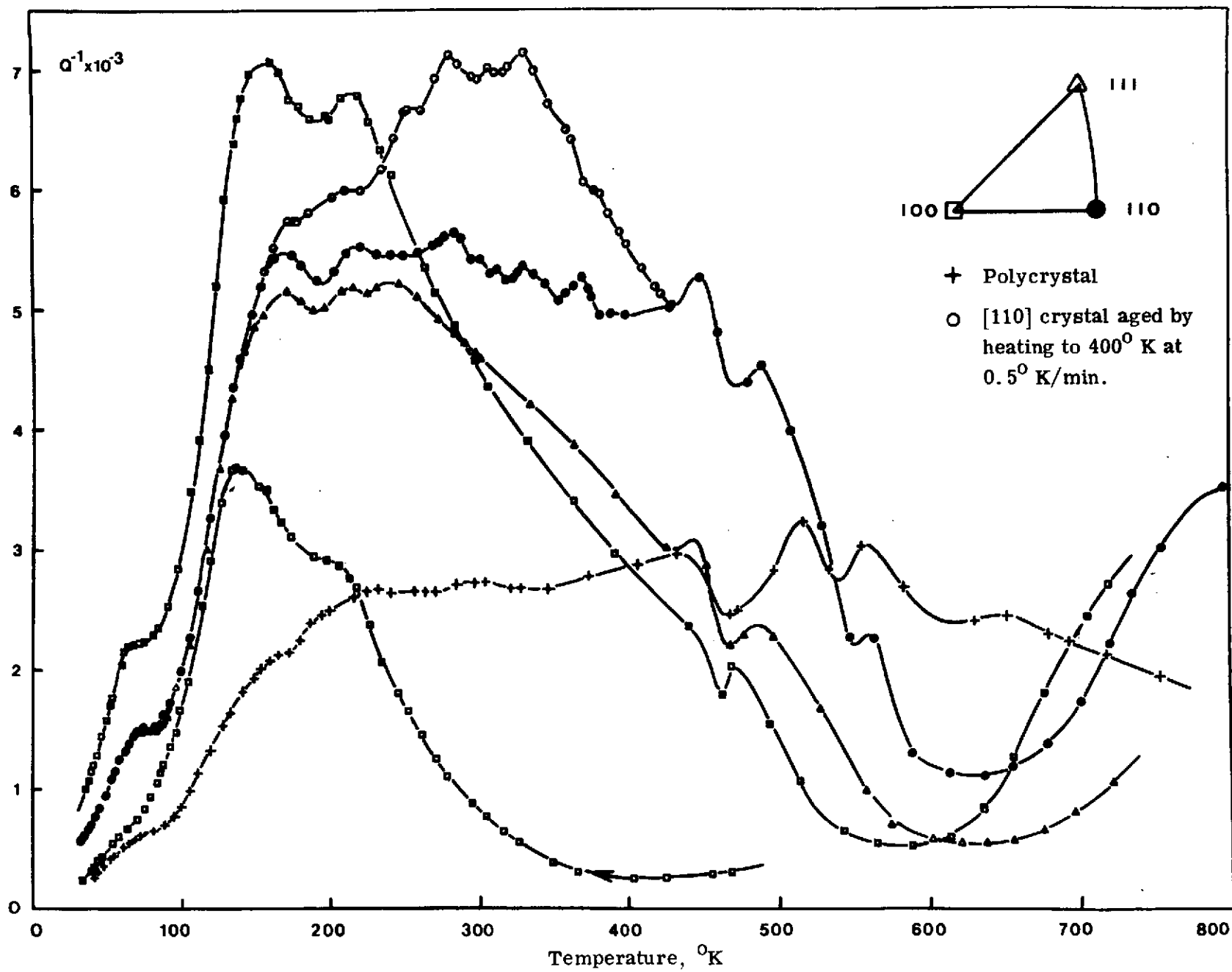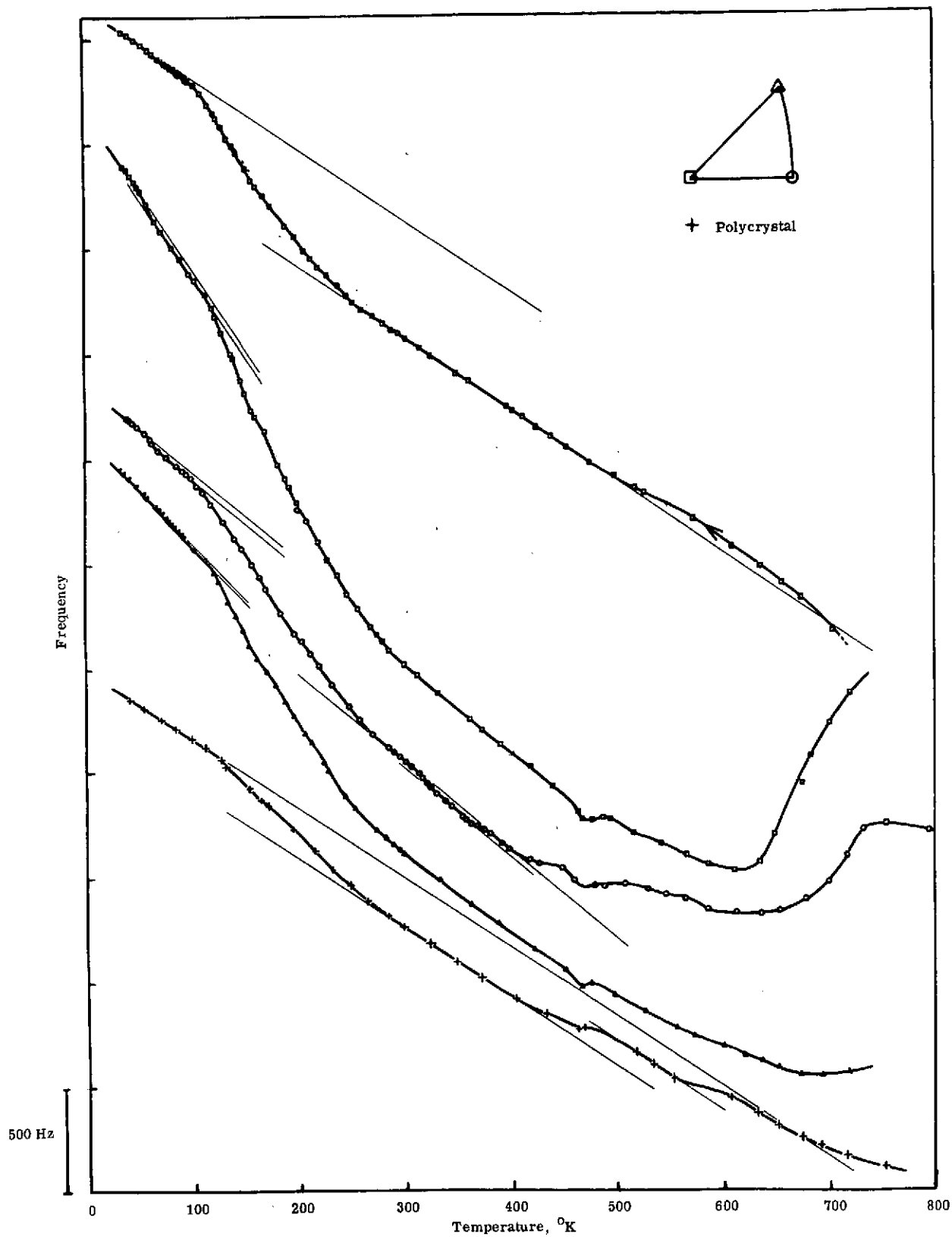
Figure 7: Cold worked samples.

Figure 8: Modulus effects.